

## Common ion effects in zeoponic substrates: wheat plant growth experiment

John E. Gruener <sup>a,\*</sup>, Douglas W. Ming <sup>b</sup>, Keith E. Henderson <sup>c</sup>,  
Charles Galindo Jr. <sup>a</sup>

<sup>a</sup> *Hernandez Engineering, Inc., 16055 Space Center Boulevard, Suite 725, Houston, TX 77062, USA*

<sup>b</sup> *NASA Johnson Space Center, Mail Code SX, Houston, TX 77058, USA*

<sup>c</sup> *NASA Johnson Space Center, Mail Code EC, Houston, TX 77058, USA*

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### Abstract

The National Aeronautics and Space Administration (NASA) is developing a zeolite-based synthetic substrate, termed zeoponics. The zeoponic substrate, consisting of  $\text{NH}_4^+$ - and  $\text{K}^+$ -exchanged clinoptilolite, and a synthetic apatite, provides all of the plant-essential nutrients through mineral dissolution and ion exchange, with only the addition of water. Initial experiments with wheat resulted in good vegetative growth, however, a subsequent experiment grown to maturity resulted in poor seed yield. Plant-tissue analyses suggested that high P and low Ca concentrations were possible reasons for the low yield. To address the nutrient imbalances, the Ca-bearing minerals dolomite, wollastonite, and calcite were examined as amendments to the zeoponic substrate to provide a common ion effect (i.e., Ca increases in solution from the dissolution of the Ca-bearing minerals will reduce the dissolution of the synthetic apatite, and thus reduce the amount of P available for plant uptake). Plant tissue Ca for all wheat plants grown in amended zeoponic substrates increased with increasing mineral solubilities of the added Ca-bearing minerals, while plant tissue P decreased with increasing mineral solubilities of the added Ca-bearing minerals. Wheat plants grown with the calcite amendment had the highest Ca concentrations (i.e., 0.45 wt.%) and the lowest P concentrations (i.e., 0.76 wt.%). Seed yield and harvest index were also highest of the plants grown in zeoponic substrate when calcite was the amendment (i.e., 1.57 g/plant and 45.7, respectively). This data illustrates that Ca-bearing minerals do provide a common ion effect in zeoponic substrates.

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### 1. Introduction

The National Aeronautics and Space Administration (NASA) is developing systems that are capable of growing plants in space. NASA is also

developing regenerative life support systems, in which plants will be relied upon to sequester carbon dioxide and generate oxygen through photosynthesis, produce potable water through transpiration, and produce food through plant growth and reproduction [1]. In regenerative life-support systems, air, water, food, and waste are all recycled and reused in an integrated fashion.

\* Corresponding author.

To provide plant-essential nutrients, aeration, moisture retention, and mechanical support for the growing plants, the Johnson Space Center (JSC) has developed a zeolite-based synthetic substrate consisting of clinoptilolite and synthetic apatite (SA) [2]. Zeoponic plant-growth systems have been defined as the cultivation of plants in artificial soils, which have zeolite as a major component [3]. In the JSC zeoponic substrate, the native clinoptilolite cations are exchanged for  $\text{NH}_4^+$  and  $\text{K}^+$  cations, [4], which then become the primary N and K sources for plant nutrition. The dynamic equilibria that takes place between zeolite and apatite in a zeoponic system are discussed in detail by Lai and Eberl [5] and Ming and Allen [6,7]. In brief, dissolution of the apatite supplies  $\text{Ca}^{2+}$  to the soil solution. Through ion-exchange reactions, solution  $\text{Ca}^{2+}$  will remove  $\text{NH}_4^+$  and  $\text{K}^+$  from zeolitic exchange sites, thus making the  $\text{NH}_4^+$  and  $\text{K}^+$  cations available for plant uptake. The goal of the zeoponic research efforts at JSC is to develop a solid substrate that will slowly deliver most of the plant-essential macronutrients (N, P, K, Ca, Mg, and S) and all of the plant-essential micronutrients (Zn, Fe, Cu, Mn, B, Mo, and Cl) for many growth seasons, with the remainder of the required macronutrients (H, O, and C) being supplied by the water and air used throughout a space habitat (Fig. 1).

Initial plant-growth experiments with wheat grown in zeoponic substrates resulted in excellent vegetative growth [2]. However, when wheat plants were grown to maturity in another experiment with zeoponic substrates, seed yield was poor [8]. Plant-tissue analyses suggested that high P and low Ca concentrations were possible reasons for the low yield [8]. An additional experiment with wheat investigating the effects of adding nitrifying bacteria, dolomite and ferrihydrite to the zeoponic substrate resulted in better yields [9]. Beiersdorfer et al. [10] described a common-ion effect that occurs in zeoponic substrates amended with Ca-bearing minerals, where the solubility of the SA is diminished by the presence of  $\text{Ca}^{2+}$  in solution from the dissolution of the Ca-bearing minerals. This resulted in higher  $\text{Ca}^{2+}$  and lower P in solution.

The purpose of the present paper is to report on a plant-growth experiment investigating the response of wheat plants grown in clinoptilolite–apatite substrates amended with three different Ca-bearing minerals. The specific objective of the experiment was to observe whether a common-ion effect described by Beiersdorfer et al. [10] would result in increased Ca plant uptake, reduced P plant uptake, and higher yields by wheat plants grown in the amended zeoponic substrates.

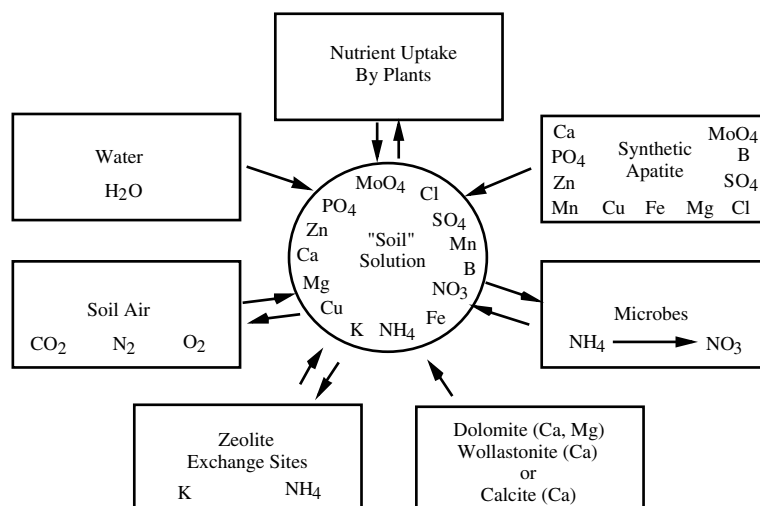


Fig. 1. Dynamic equilibria for NASA's zeoponic plant-growth system. Plant growth nutrients are slowly released from synthetic apatite (SA) and Ca-bearing amendments by dissolution and from clinoptilolite (Cp) by ion-exchange reactions.

## 2. Experimental

### 2.1. Materials

Clinoptilolite-rich tuff (Cp) from the Fort LeClède deposit, Sweetwater County, Wyoming and a synthetic hydroxyapatite produced in the laboratory were used as the primary components of the zeoponic substrate. The Fort LeClède clinoptilolite is nearly mono-mineralic as determined by X-ray diffraction analysis [11], with only trace amounts of quartz present. The Cp was transformed into either an ammonium-exchanged ( $\text{NH}_4\text{-Cp}$ ) or a potassium-exchanged ( $\text{K-Cp}$ ) form, using the method of Allen et al. [4]. The  $\text{NH}_4^+$  and  $\text{K}^+$  cation-exchange capacity (CEC) of the clinoptilolite-rich tuff were 208 and 191 meq/100 g, respectively, determined by a CsCl method described by Ming and Dixon [12].

The SA is an agronutrient-substituted hydroxyapatite, produced in the laboratory according to the method of Golden and Ming [13]. In addition to Ca and  $\text{PO}_4$ , the SA has Mg,  $\text{SO}_4$ , and micronutrients (i.e., Zn, Fe, Cu, Mn, B, Mo, and Cl) incorporated in its structure. The chemical composition of the SA is shown in Table 1.

The three Ca-bearing minerals chosen to be used as amendments to the zeoponic substrate were dolomite (Baker Grandol Regular #4, Baker Refractories, York, Pennsylvania, USA), wollastonite, (Lewis mine, NYCO Minerals Inc., New York, USA), and calcite (D. J. Minerals M-61, Montana, USA). The mean chemical compositions

Table 1

Chemical composition (wt.%) of the synthetic apatite (SA) used in the zeoponic substrates

Major oxides <sup>a</sup>	Wt.%
CaO	46.6
$\text{P}_2\text{O}_5$	34.6
MgO	2.77
$\text{SiO}_2$	2.56
$\text{SO}_3$	2.46
$\text{Fe}_2\text{O}_3$	1.76
ZnO	0.06
MnO	0.06
CuO	0.02

<sup>a</sup> Hydroxyapatite produced in the laboratory by the method of Golden and Ming [13].

for the three Ca-bearing minerals are shown in Table 2, as measured by Beiersdorfer et al. [10]. All materials, (i.e.,  $\text{NH}_4\text{-Cp}$ ,  $\text{K-Cp}$ , SA, dolomite, wollastonite, and calcite) were crushed and sieved to a particle size of 0.5–1.0 mm.

### 2.2. Methods

The zeoponic materials and the Ca-bearing minerals were combined to form three zeoponic substrates, each consisting of 36 wt.%  $\text{NH}_4\text{-Cp}$ ; 36 wt.%  $\text{K-Cp}$ ; 18 wt.% SA; and 10 wt.% dolomite, wollastonite, or calcite. Two control substrates were also developed: a potting mix control composed of peat, vermiculite, and perlite in a 1:1:1 volume ratio, and a K-exchanged Cp ( $\text{K-Cp}$ ) control. The five substrates (i.e., Cp-SA-dolomite, Cp-SA-wollastonite, Cp-SA-calcite, potting mix

Table 2

Mean chemical composition (wt.%) of the dolomite, wollastonite, and calcite used in the zeoponic substrates (after Beiersdorfer et al. [10])

Major oxides	Dolomite	Wollastonite	Calcite
MgO	17.66 (0.12) <sup>a</sup>	—	—
CaO	35.03 (0.15)	48.37 (0.19)	55.72 (0.05)
MnO	0.02 (0.03)	0.19 (0.03)	0.25 (0.04)
FeO	0.31 (0.08)	0.49 (0.07)	0.09 (0.03)
$\text{CO}_2$	46.98 (0.02)	—	43.94 (0.01)
$\text{Al}_2\text{O}_3$	—	0.03 (0.02)	—
$\text{SiO}_2$	—	51.23 (0.14)	—
Total	100.0 (0.00)	100.32 (0.23)	100.0 (0.00)

<sup>a</sup> Numbers in parentheses represent one standard deviation (for dolomite,  $n = 25$ ; for wollastonite,  $n = 25$ ; for calcite,  $n = 28$ , where  $n = \#$  of analyses).

control, K–Cp control) were placed in pots with dimensions of 9.5 cm in diameter and 8.5 cm deep. The substrate volume in each pot was 440 ml and the average bulk density of the zeoponic substrates was 1 g/cm<sup>3</sup>. Thus, each pot contained 440 g of zeoponic materials, or 440 ml of potting mix. The zeoponic substrates were inoculated with *Nitrosomonas* and *Nitrobacter* bacteria to establish a nitrification process, in which the NH<sub>4</sub><sup>+</sup> released from the NH<sub>4</sub>–Cp is oxidized to form NO<sub>3</sub><sup>–</sup>.

‘USU-Apogee’, a full-dwarf hard red spring wheat (*Triticum aestivum* L.) cultivar developed for high yields in controlled environments [14] was grown in the zeoponic and control substrates for 90 days. Four replications were used for each of the five substrate treatments. The total experiment consisted of 20 pots arranged in a randomized complete block design. The zeoponic substrates were watered with deionized water throughout the experiment, and the control substrates were watered with 1/2-strength Hoagland’s nutrient solution [15].

Wheat seeds were soaked overnight in water and planted, 20 seeds per pot. After emergence, plants were thinned to 15 per pot. The wheat was grown in an environmental growth chamber (Environmental Growth Chambers, Chagrin Falls, Ohio, Model G-15) under the following average daily conditions: 16 h of daylight at 25 °C, 8 h of darkness at 23 °C; 55–85% relative humidity, and a photosynthetic photon flux that averaged 500 μmol m<sup>–2</sup> s<sup>–1</sup>. All treatments were irrigated to excess six times daily with an automated watering system at an average rate of 20 ml/min, for durations of 32 s per irrigation at the beginning of the experiment to 112 s per irrigation at peak plant water usage. Five shoots were harvested from each pot at 30 days after planting and the remaining 10 shoots in each pot were harvested at 90 days. Oven-dried (70 °C for 48 h) plant samples were weighed to determine the dry-matter production. Dried plant samples were sent to The University of Wisconsin at Madison soil and plant analysis laboratory for N, P, K, Ca, Mg, S, Zn, Fe, Cu, Mn, B, Mo, and Cl plant-tissue analysis. Data was analyzed using an analysis of variance (ANOVA), and significant differences were compared using a Duncan’s multiple range test. The test is based on

ordering the means from smallest to largest and computing all the differences (all differences between means). The differences are then compared to a least significant range. If a difference exceeds the least significant range value, then the result is significant.

### 3. Results and discussion

#### 3.1. Dry-matter production

Dry matter was harvested when the wheat plants were at 90 days of growth. Dry-matter production for wheat grown in Cp–SA-dolomite and Cp–SA-calcite substrates was equivalent to or higher than the dry-matter production for wheat grown in the control substrates, while dry-matter for wheat grown in Cp–SA-wollastonite was less than the dry-matter production for wheat grown in the control substrates (Table 3). The average dry-matter production for plants grown in Cp–SA-dolomite, Cp–SA-wollastonite, and Cp–SA-calcite was 3.14, 2.91, and 3.44 g/plant, respectively. The average dry-matter production for wheat plants grown in the potting mix and K–Cp controls was 3.19 and 3.12 g/plant, respectively. Statistically, there were no significant differences in dry-matter production among all substrates. The zeoponic dry-matter production in this experiment was higher than the 2.71 g/plant reported by Henderson et al. [9] for wheat plants grown in a Cp–SA-dolomite substrate similar to the Cp–SA-dolomite substrate used in this experiment.

#### 3.2. Wheat-seed production

Wheat seed was collected from plants harvested at 90 days of growth. The wheat-seed production for plants grown in all zeoponic substrates was less than the wheat-seed production of plants grown in the control substrates (Table 3). The average wheat-seed production for plants grown in Cp–SA-dolomite, Cp–SA-wollastonite, and Cp–SA-calcite was 1.40, 1.28, and 1.57 g/plant, respectively. The average wheat-seed production for wheat plants grown in the potting mix and K–Cp controls was 1.70 and 1.59 g/plant, respectively.

Table 3

Average dry-matter and wheat-seed production and above ground harvest index for 'USU-Apogee' wheat grown in zeoponic substrates and control substrates for 90 days

	Zeoponic substrate w/dolomite	Zeoponic substrate w/wollastonite	Zeoponic substrate w/calcite	K-exchanged clinoptilolite control	Potting mix control
Average dry-matter per plant <sup>a</sup> (g)	3.14 <sup>b</sup> (0.37) <sup>c</sup>	2.91 (0.44)	3.44 (0.37)	3.12 (0.20)	3.19 (0.32)
Average seed yield per plant (g)	1.40 (0.25)	1.28 (0.17)	1.57 (0.16)	1.59 (0.15)	1.70 (0.20)
Average harvest index per plant <sup>c</sup>	44.2 <sup>d</sup> (4.26)	44.1 <sup>d</sup> (1.39)	45.7 <sup>d</sup> (0.78)	51.0 <sup>e</sup> (2.04)	53.4 <sup>e</sup> (1.80)

<sup>a</sup> Includes above ground dry-matter only.

<sup>b</sup> Data for wheat plants at 90 days of growth.

<sup>c</sup> Numbers in parentheses represent one standard deviation ( $n = 4$ ).

<sup>d,e</sup> Means in a row with different superscripts differ significantly ( $P < 0.05$ ).

Statistically, there were no significant differences in wheat-seed production among all substrates. However, the results from this experiment do show yield improvements over past zeoponic wheat growth experiments. Henderson et al. [9] reported a yield of 1.2 g/plant for wheat plants grown in a Cp-SA-dolomite, inoculated with nitrifying bacteria, and Gruener et al. [8] reported yields averaging 0.77 g/plant over three crops grown in Cp-SA substrates without a Ca-bearing amendment or an inoculation with nitrifying bacteria.

The lower wheat-seed production in plants grown in zeoponic substrates resulted in lower harvest index values as well (Table 3). The average harvest index values (using above ground dry matter only) for wheat plants grown in Cp-SA-dolomite, Cp-SA-wollastonite, and Cp-SA-calcite were 44.2, 44.1, and 45.7, respectively. Wheat plants grown in the potting mix control substrate had the highest average harvest index, at 53.4,

while the K-Cp grown wheat plants had an average harvest index of 51.0. There were no significant differences in the harvest index values among the plants grown in the zeoponic substrates amended with the three different Ca-bearing minerals. There were also no significant differences in the harvest index values among the plants grown in the control substrates. However, there were significant differences in harvest index values between plants grown in zeoponic substrates and plants grown in control substrates.

### 3.3. Plant-nutrient analysis

Average plant-tissue concentrations of the macronutrients (N, P, K, Ca, Mg, S) and the micronutrients (Zn, Fe, Cu, Mn, B, Mo, Cl) are listed in Tables 4 and 5, respectively, for 28-day old wheat plants grown in zeoponic and control substrates. Average N concentrations of plants grown

Table 4

Average plant-tissue analyses for macronutrients (wt.%) for 'USU-Apogee' wheat grown in zeoponic substrates and control substrates

	Zeoponic substrate w/dolomite	Zeoponic substrate w/wollastonite	Zeoponic substrate w/calcite	K-exchanged clinoptilolite control	Potting mix control
Nitrogen	5.62 <sup>a</sup> (0.34) <sup>b</sup>	5.65 (0.20)	5.29 (0.16)	4.25 (0.33)	4.64 (0.19)
Phosphorus	1.02 (0.04)	0.90 (0.05)	0.76 (0.11)	0.63 (0.03)	0.82 (0.03)
Potassium	4.78 (0.15)	4.29 (0.33)	4.05 (0.38)	4.86 (0.16)	4.68 (0.28)
Calcium	0.23 (0.05)	0.28 (0.01)	0.45 (0.15)	0.17 (0.01)	0.36 (0.02)
Magnesium	0.29 (0.05)	0.22 (0.01)	0.17 (0.04)	0.20 (0.02)	0.31 (0.01)
Sulfur	0.40 (0.01)	0.35 (0.02)	0.34 (0.03)	0.34 (0.02)	0.54 (0.06)

<sup>a</sup> Data for wheat plants at 28 days of growth.

<sup>b</sup> Numbers in parentheses represent one standard deviation ( $n = 4$ ).

Table 5

Average plant-tissue analyses for micronutrients (mg/kg) for 'USU-Apogee' wheat grown in zeoponic substrates and control substrates

	Zeoponic substrate w/dolomite	Zeoponic substrate w/wollastonite	Zeoponic substrate w/calcite	K-exchanged clinoptilolite control	Potting mix control
Zinc	29.3 <sup>a</sup> (1.53) <sup>b</sup>	28.8 (0.71)	29.5 (2.04)	38.3 (2.36)	51.6 (2.55)
Iron	104 (36.3)	84.5 (4.64)	84.8 (6.15)	78.2 (3.60)	87.8 (7.30)
Copper	12.4 (0.24)	11.8 (1.26)	10.1 (0.77)	7.41 (0.73)	13.3 (1.28)
Manganese	33.7 (2.79)	45.4 (2.22)	63.2 (2.58)	81.8 (7.91)	203 (25.5)
Boron	19.2 (1.95)	15.6 (1.04)	11.5 (3.95)	9.67 (1.65)	12.1 (3.39)
Molybdenum	0.31 (0.07)	0.34 (0.47)	0.04 (0.06)	0.00 (0.00)	0.23 (0.41)
Chlorine	633 (79.9)	611 (57.4)	694 (95.2)	1954 (40.9)	2270 (169)

<sup>a</sup> Data for wheat plants at 28 days of growth.<sup>b</sup> Numbers in parentheses represent one standard deviation ( $n = 4$ ).

in zeoponic substrates ranged from 5.29 to 5.65 wt.%, which are at the upper end or above the typical range of 3.0–5.5 wt.% for winter wheat grown in field conditions, as measured by Karlen and Whitney [16]. The average N concentrations of the control plants were within the typical range. Average K concentrations of plants grown in zeoponic substrates ranged from 4.04 to 4.78 wt.%, which are at the upper end or above the typical range of 2.0–4.5 wt.% for K in wheat, as measured by Karlen and Whitney [16]. The average K concentrations of the control plants were above the typical range. The results for the N and K concentrations in plants grown in zeoponic substrates were unexpected, particularly the result that the lowest concentrations of N and K occurred in plants grown in Cp-SA-calcite. It was thought that the plants grown in Cp-SA-calcite would have the highest N and K concentrations, since calcite is the most soluble of the three Ca-bearing amendments and would provide more  $\text{Ca}^{2+}$  cations available for exchange with  $\text{NH}_4^+$  and  $\text{K}^+$  on zeolitic exchange sites. It is possible the larger amounts of  $\text{Ca}^{2+}$  ions in solution inhibited the plants ability to take up  $\text{NH}_4^+$  and  $\text{K}^+$ .

Average P concentrations of plants grown in both the zeoponic and control substrates were higher than the typical range of 0.3–0.6 wt.% for wheat, as measured by Karlen and Whitney [16]. The P concentration of plants grown in Cp-SA-dolomite had the highest value at 1.02 wt.%, while the P values for wheat grown in Cp-SA-wollastonite and Cp-SA-calcite were 0.90 and 0.76 wt.%,

respectively. There is a direct correlation between plant P concentrations and the Ca-bearing minerals' solubilities. Plants grown in zeoponic substrates amended with calcite, the most soluble of the three Ca-bearing minerals, had the lowest P concentrations of wheat grown in zeoponic substrates. Plants grown in zeoponic substrates amended with dolomite, the least soluble of the three Ca-bearing minerals, had the highest P concentrations. This suggests that the solubilities of the Ca-bearing amendments had a direct effect on the dissolution rate of the SA, and thus the available P for plant uptake. The average P concentrations reported in this experiment are much lower than the 1.97 wt.% P concentrations that Gruener et al. [8] measured in first-crop wheat grown in zeoponic substrates without any Ca-mineral amendment.

Average Ca concentrations for plants grown in zeoponic substrates and the potting mix control substrate were within the typical range for wheat plants of 0.2–0.55 wt.%, as measured by Karlen and Whitney [16]. Calcium concentrations for plants grown in the K-Cp control substrate were lower, with a value of 0.17 wt.%. The Ca concentration of plants grown in Cp-SA-calcite had the highest value at 0.45 wt.%, while the Ca values for Cp-SA-wollastonite and Cp-SA-dolomite grown plants were 0.28 and 0.23 wt.%, respectively. There is a direct relationship between plant Ca concentrations and available Ca in the soil water for plant uptake. The plants grown in zeoponic substrate amended with calcite, the most soluble of the three

Ca-bearing minerals, had the highest Ca concentrations of wheat grown in zeoponic substrates. The plants grown in zeoponic substrate amended with dolomite, the least soluble of the three Ca-bearing minerals, had the lowest Ca concentrations.

Average Mg concentrations for plants grown in zeoponic substrates and the potting mix control substrate were above or within the typical range of 0.1–0.25 wt.% for Mg in wheat plants, as measured by Karlen and Whitney [16]. The Mg concentration of 0.29 wt.% in plants grown in Cp-SA-dolomite was the highest Mg value for wheat grown in the amended zeoponic substrates. This is due to dolomite being the only amendment with Mg in its crystalline structure, and thus available for plant uptake upon mineral dissolution. The Mg concentrations for plants grown in Cp-SA-wollastonite and Cp-SA-calcite were 0.22 and 0.17 wt.%, respectively. This correlates with calcite being the more soluble of the two minerals, which further slows the dissolution of the SA and further reduces the Mg available for plant uptake.

Plant tissue concentrations of S and the micronutrients were supplied at the expected levels. Most of these nutrients show trends similar to that discussed for P. Nutrient concentrations are generally highest in plants grown in zeoponic substrates amended with dolomite, the least soluble mineral of the three Ca-bearing amendments, and lowest in plants grown in zeoponic substrates amended with calcite, the most soluble mineral of the three Ca-bearing amendments. This suggests that the SA's dissolution was directly affected by the solubilities of the Ca-bearing minerals, with the solubility of the SA depressed by the largest amount by the addition of the most soluble amendment (i.e., calcite).

#### 4. Conclusions

This results from this experiment suggest that Ca-bearing minerals do provide a common ion effect (i.e., Ca) when added to zeoponic substrates, increasing the available Ca in soil solution for plant uptake, while decreasing the dissolution of the SA, and thus reducing the available P for plant

uptake. Zeoponic substrates amended with Ca-bearing minerals can provide all of the plant-essential nutrients in the proper concentrations for wheat plants, with only the addition of water, resulting in excellent seed yield. NASA's zeoponic substrate has been successfully flown on Space Shuttle missions [17], and shows promise as a long-term nutrient supply for plant-growth experiments on the International Space Station.

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